TRANSPORTATION POOLED FUND PROGRAM
QUARTERLY PROGRESS REPORT

Lead Agency (FHWA or State DOT): Oklahoma Department of Transportation

INSTRUCTIONS:
Project Managers and/or research project investigators should complete a quarterly progress report for each calendar quarter during which the projects are active. Please provide a project schedule status of the research activities tied to each task that is defined in the proposal; a percentage completion of each task; a concise discussion (2 or 3 sentences) of the current status, including accomplishments and problems encountered, if any. List all tasks, even if no work was done during this period.

Transportation Pooled Fund Program Project # *TPF-5(297)*	**Transportation Pooled Fund Program - Report Period:** X Quarter 1 (January 1 – March 31) O Quarter 2 (April 1 – June 30) O Quarter 3 (July 1 – September 30) O Quarter 4 (October 1 – December 31)	
Project Title: **Improving Specifications to Resist Frost Damage in Modern Concrete Mixtures**		
Name of Project Manager(s): **Tyler Ley**	**Phone Number:** **405-744-5257**	**E-Mail** Tyler.ley@okstate.edu
Lead Agency Project ID: **TPF-TPF5(297)RS / JOB PIECE 30802(04)**	**Other Project ID (i.e., contract #):** AA-5-52974	**Project Start Date:** March 10, 2014
Original Project End Date: February 28, 2017	**Current Project End Date:**	**Number of Extensions:**

Project schedule status:

On schedule ☐ On revised schedule ☐ Ahead of schedule X Behind schedule

Overall Project Statistics:

Total Project Budget	Total Cost to Date for Project	Percentage of Work Completed to Date
$572,500	$190,000	30%

Quarterly Project Statistics:

Total Project Expenses and Percentage This Quarter	Total Amount of Funds Expended This Quarter	Total Percentage of Time Used to Date
$40,000	$40,000	33%

Project Description:

Concrete can be damaged when it is 1) sufficiently wet (has a high degree of saturation) and 2) is exposed to temperature cycles that enable freezing and thawing. The damage that occurs due to freezing and thawing can lead to premature deterioration, costly repairs, and premature replacement of concrete infrastructure elements. Current specifications for frost durability are largely based on work completed in the 1950s, and while this work included many landmark discoveries (Kleiger 1952, 1954) it may not be completely representative of materials used in modern concrete mixtures. Results from recent studies suggest that there are several ways in which frost damage can be reduced through new tests and improve specifications that can lead to extended service life of concrete infrastructure.

The goal of the research is to produce improved specifications, and test methods; while, improving the understanding of the underlying mechanisms of frost damage. Specifically, this work will seek to develop new test procedures that may be faster and/or more reliable than the existing methods. The objectives of this project are:

- Determine the necessary properties of the air-void system to provide satisfactory frost durability in laboratory testing of laboratory and field concretes with different combinations of admixtures, cements, and mixing temperatures
- Determine the accuracy of a simple field test method that measures air void system quality with field and laboratory concrete
- Determine the critical combinations of absorption and the critical degree of saturation on the frost durability in accelerated laboratory testing
- Establish new test methods and specifications for fresh and hardened concrete to determine frost durability and field performance

In addition a streaming lecture series on freeze-thaw durability will be generated as part of this work.

Progress this Quarter (includes meetings, work plan status, contract status, significant progress, etc.):

Task 1: Literature Review and Development of the Testing Matrix (OSU and Purdue)

In this task the research teams will review the existing literature and determine a testing matrix to cover the necessary variables. Work is needed to understand how cements of different alkali content, different mixing temperatures, and types of mixing impact the air entrainment system and subsequently the frost durability of concrete. These variables can lead to changes in AEA effectiveness and their impact needs to be quantified with ASTM C 666 testing. As part of this task we will work with our project oversight committee to establish a set of materials and a testing matrix that can be used for the entire study. The decisions used in developing this test matrix will be made based on literature review, previous research by the PIs and the needs identified by the study advisory discussions.

The initial testing matrix will focus on understanding the impact of different air void systems on the freeze thaw durability of concrete. The matrix has been developed and was shared with the research oversight committee in the first conference call. Plans for future investigations were discussed in the second conference call. The team first looked at mixtures for pavements and then moved to mixtures for pavements. Here is an overview of the mixtures:

Limestone aggregate and natural sand from Oklahoma will be used for these mixtures. Both aggregates have been shown great freeze thaw field performance in the laboratory and the field. A mixture with 20% class C fly ash will be investigated with 6.5 sacks of total cementitious content and a w/cm of 0.45. A wood rosin AEA will be the primary admixture investigated as it is the most widely used AEA. However, select mixtures will be investigated with synthetic AEA. These AEAs will be used to produce mixtures with different spacing factors and air content. These samples will be investigated in a number of the freeze thaw tests in the project. Next, a mixture with 0.40 w/cm will be investigated with the same AEA. After that mixtures with high range water reducers will be investigated with 0.40 and 0.35 w/cm. A few mixtures with different high range water reducer dosages will be investigated.

On this project over 80 concrete mixtures have been investigated. Work is still ongoing to complete the hardened air void analysis and the rapid freeze thaw testing. In the next year mixtures with different admixture combinations will be investigated as well as the impact of different construction methods on the quality of the air void system. The team will also investigate how the SAM needs to be modified for use with light weight aggregate.

85% complete (This task will not reach 100% until the end of the project as changes are continually made to the testing matrix.)

Task 2: Sample Preparation (OSU and Purdue)

A number of the mixtures suggested in task 1 have been completed at OSU and have been shipped to Purdue for testing. Mixtures with different amounts of superplasticizers and air entraining agents have been investigated. Mixtures are also being complete to look at different dosages of superplasticizer and also some mid-range water reducers. Over 80 mixtures have been investigated. In the next quarter mixtures will be investigated that use different combinations of admixtures. The impact of different construction procedures on the quality of the air void system will also be investigated. The test method will also be modified to allow the usage of light weight aggregate.

The research team also plans to hold a precision and bias testing at OSU in the winter. For this testing we will invite experienced users of the SAM to come to Oklahoma and all use their equipment to investigate a single concrete mixture. This will generate important data for the precision and bias statement of the AASHTO test method.

45% complete

Task 3: Validation of the Super Air Meter (OSU)

In this task the Super Air Meter (SAM) will be evaluated in laboratory and field mixtures. The laboratory mixtures to be investigated include: aggregate with high aggregate correction factor, light weight aggregate, hot weather concrete, cold weather concrete, and any other items that the research oversight committee feels is important. In addition a number of mixtures will be investigated in the field. This will be done by visiting local ready mix and central mix batch plants to take samples.

Over 80 laboratory mixtures have been completed and the results are being complied. The data shows that the SAM does a good job of predicting the spacing factor for the majority of the mixtures investigated. The SAM limit of 0.20 psi has shown a conservative correlation to a spacing factor of 0.008" 94% of the time. For all of these mixtures at least two different SAMs are being investigated in order to collect the precision and bias information needed for the AASHTO test method. Work will be done to look at different dosages of high range water reducer and the impact on the air content, SAM number, and spacing factor relationship. Work is continuing to be completed to investigate the variability in the test method. The current results are shown in Fig. 1.

In addition, the research team has evaluated over 70 different field concrete mixtures. For all of these mixtures we have used two SAMs to evaluate each mixture. We plan to continue to add measurements. These results are shown in Fig. 2.

The Turner Fairbanks Lab at FHWA has also completed a 3rd party evaluation of the SAM and they have provided their data for comparison. The results are shown in Fig. 3.

The results from the lab, field, and 3rd party evaluations are included in Figure 4. In this report. The data continues to be very consistent and show that there is about a 93% agreement between spacing factor recommendation of 0.008" and a SAM number of 0.20.

In addition commercial versions of the SAM have been provided to the following partners for their usage: Purdue, Iowa, Nebraska, Kansas, North Dakota, Illinois, Oklahoma, Pennsylvania, Minnesota, and Wisconsin. The following states have either already provided or have agreed to supply field data: Iowa, Michigan, Wisconsin, Minnesota, North Dakota, Kansas, Nebraska, Colorado, and Utah. In addition the FHWA mobile concrete lab has supplied samples from Pennsylvania, Tennessee, North Dakota, and Florida. These samples are being prepared and polished for hardened air-void analysis. This data will contribute to the vast data set that is being compiled. So far there is over 70% agreement between the field data and the SAM limit of 0.20. There seems to better agreement with a SAM limit of 0.25; however, the 0.20 limit is conservative.

45% complete

Task 4: Creation of an AASHTO Test Method and Specification for the SAM (OSU)
A presentation was made to the AASHTO Materials subcommittee. The presentation was well received and we have submitted the provisional test method for publication. Several revisions to the test method were made but the final version has been submitted for publication. This is a great accomplishment!

Work still needs to be done to update the provision and bias statement for the test method. Work is also being done to add the usage of light weight aggregate to the test method.

55% complete

Task 5: Use of X-Ray Tomography of Air Voids and Frost Damage (OSU)

Researchers at OSU have developed nondestructive techniques to examine microscopic air voids in fresh and hardened concrete by using a X-ray micro computed tomography (mCT) scanner. This is a powerful technique that allows measurements to be made not previously possible. The research team has developed techniques to image water movements and have access to a freezing stage. By combining this information about the void distribution, the moisture content and distribution, and then being able to image the damage that occurs from freezing is a powerful tool. These observations can lead to ground breaking insights into the mechanisms of frost damage and how it can be avoided.

The experimental methods are largely finished. Some preliminary work is still being done to investigate the method.

Purdue provided 12 samples that the team at OSU scanned at a one micron resolution and then sent back to Purdue so that they could investigate them in the neutron beamline at NIST. In the beamline the samples were flooded with water and then freezing cycles were used to try and create damage in the samples. The samples will be sent back to OSU and then they will be scanned again to see if any cracks can be observed.

Progress 20%

Task 6: ASTM C 666 (OSU)

The primary test method used to investigate the frost durability of the concrete will be the ASTM C 666 test. This test is the most widely recognized test to investigate the rapid deterioration from freezing and thawing. As many mixtures will be investigated with this test as possible. As part of this task the specimen absorption and desorption of the samples will be investigated using a modified form of ASTM C 1585. The impact of wetting and drying will also be investigated. While the team realizes that the ASTM C 666 is a well-respected test they feel that the three months required to complete the test is too long. The research team plans on using this information to help find a shorter test with the same rigor.

A new chamber has been purchased and a significant amount of C666 testing has been completed. A summary of the test results are shown in Fig. 5. These results show an 80% agreement with a SAM number of 0.20 and an 89% agreement if a SAM number of 0.25 is used. This difference was discussed on the conference call and a recommendation was made to stay with the 0.20 limit as it was conservative.

The research team at OSU has moved to a new laboratory. This delayed some of the ASTM C 666 testing. Work is underway to check the calibration on the chambers and get them started again.

Progress 45%

Task 7: Absorption and Desorption (Purdue)

During this task the research team will perform desorption/sorption analysis on selected mixtures prepared in Task 2. For the sorption tests 100 mm diameter samples will be used that are 50 mm in thickness. The samples will then be placed in fluid according to a modified version of ASTM C 1585 to determine the degree of saturation over time. In addition, the complete degree of saturation will be determined using vacuum saturation.

The sampling and testing protocols have been developed (as illustrated in Fig. 6) and the samples are conditioned at 50% and 75% relative humidity for the sorption tests. The 75% RH samples from the first series of samples (4 total series of samples were prepared) have reached equilibrium and testing has initiated. The samples in the other series and at 50% RH will be tested in the next two quarters. The results of this test will be used to determine the S_n value of sorption (the nick point between initial and secondary sorption) and the slope of the secondary sorption curve, which are essential for predicting long term performance. The drying test has been initiated for the first data set and the results are in the analysis stage. Testing of the different the degree of saturation at 50%, 75%, 93%, and 100% relative humidity will be performed.

Progress 30%

Task 8: Degree of Saturation and Damage Development (Purdue)

Samples prepared in Task 2 will be saturated to different degrees of saturation and the freeze-thaw tests will be performed with the samples in a sealed condition. Freeze-thaw tests will be performed on samples with 50 mm thickness and 68 mm diameter using a new Longitudinal Gaurded Comparitive Calorimeter (LGCC) setup with acoustic emissions sensing to detect damage. This test setup is capable of measuring temperature throughout the setup height to determine heat flow through the sample during each freeze-thaw cycle. Additionally, the setup is equipped with 2 acoustic emission sensors which can passively detect acoustic events as well as actively measure pulse velocity across the sample. The changes in pulse velocity throughout the freeze-thaw process can then be directly correlated to damage development in the concrete. Results from this test will be used to identify the critical degree of saturation with the express purpose of relating the critical degree of saturation to the quality of the entrained air system (for example the air void spacing). Information from this test will be used in conjunction with the results from Task 7 to determine if the air void system alters the time required to reach a critical degree of saturation (which is hypothesized with a higher SAM number corresponding to a lower S_{CRIT}). Additionally, a series of 3 cylinders will be used to determine the resistivity and degree of saturation over time of samples submerged in pore solution (to prevent alkali leaching). Additional resistivity tests will be performed at various ages and degrees of saturation on samples from a variety of tests in order to determine if a relationship exists to correlate resistivity and sorption.

The testing protocols have been developed (See Figure 6). DOS cylinders from batches 1 and 2 (20 mixtures of 34 total received by Purdue) have been tested and the results have been used to determine the degree of saturation for cylinders in the corresponding resistivity tests. Both short and long term resistivity tests have concluded for the first two batches of cylinders. These results are being analyzed to study the resistivity, formation factor, and degree of saturation over time. Experimental nick point values are being compared to theoretical values and are being related to both air volume and SAM numbers. Results to date are as expected, however conclusions will be more clear following the final round of testing, which is currently in progress and will be complete mid-May.

Batches 1 and 2 Freeze-Thaw samples have been cut and cored and are in the final stages of preparation for the LGCC (Figure 7b). A full testing schedule has been created to capture samples with different air contents and varying SAM numbers from all 3 batches (Table 1). Two LGCC setups are being utilized for testing simultaneously and both are showing good results. To date, 5 mixtures (of 11 currently planned for testing) have undergone the full spectrum of testing in the LGCC, which includes 3 degrees of saturation for each mix (approximately 100%, 95%, and 90%). Each sample is exposed to three freeze-thaw cycles over the course of five days, with pulse velocity measurements taken hourly. Results from these tests have been analyzed; however additional mixtures are necessary to draw conclusions.

Progress 30%

Task 9: Rate of Damage Analysis (Purdue)

This task will combine acoustic emission data and X-ray mCT to detect cracking and also image the location. This will be done in samples with different quality of air void systems and with different paste quality and saturation level.

Samples have been prepared for work at the NIST neutron center. Samples were prepared, x-ray tomography was performed at OSU and the samples will be imaged at NIST before and after freezing. The samples will then be returned to OSU for x-ray tomography.

Progress 10%

Task 10: Technology Transfer (OSU and Purdue)

A portion of this project will be dedicated to development of a strong educational technology transfer program. The PI's propose the development of a short course that utilizes streaming video (and could be placed on a DVD for widespread dissemination). No progress has been made on this task. This will be completed late in the project so that the latest findings can be shared with the audience.

Progress 0%

Task 11: Final Report (OSU and Purdue)

This task will be completed in the final quarter of the project.

Progress 0%

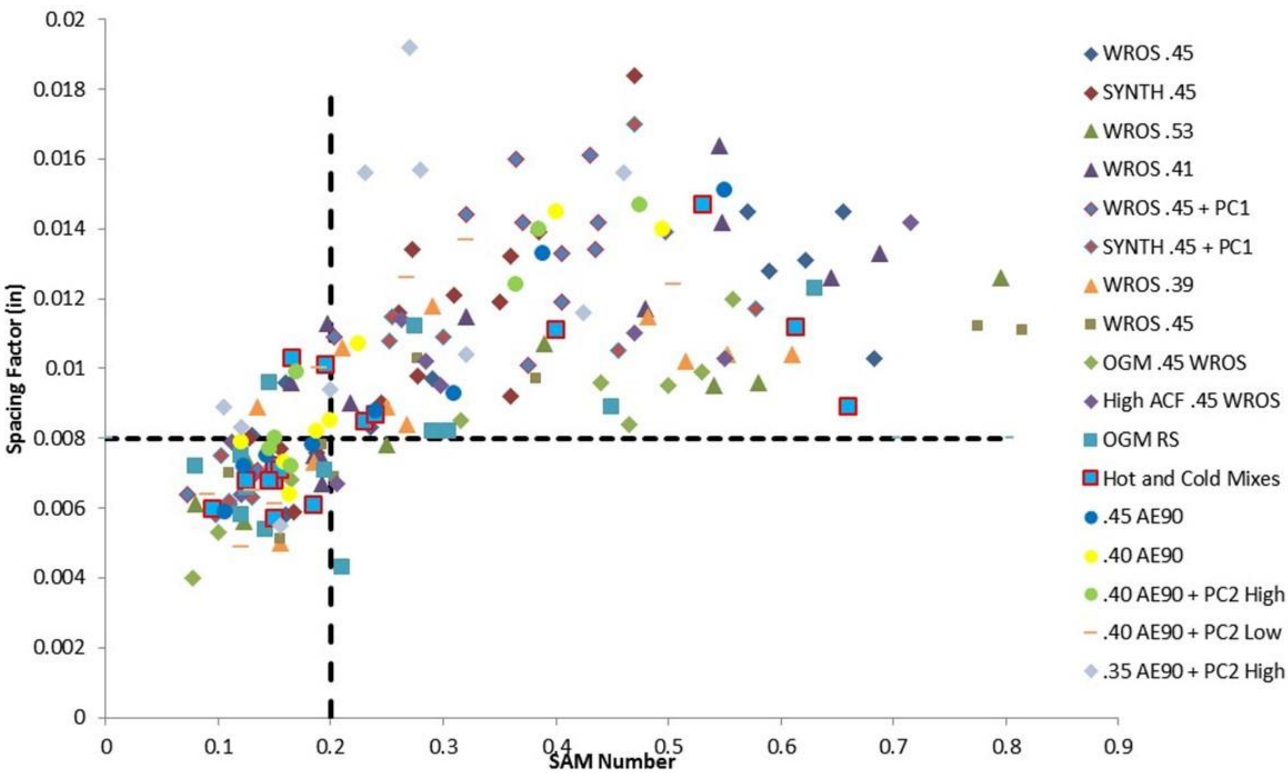


Figure 1 – Laboratory study results comparing the SAM number to the spacing factor for mixtures completed at OSU.

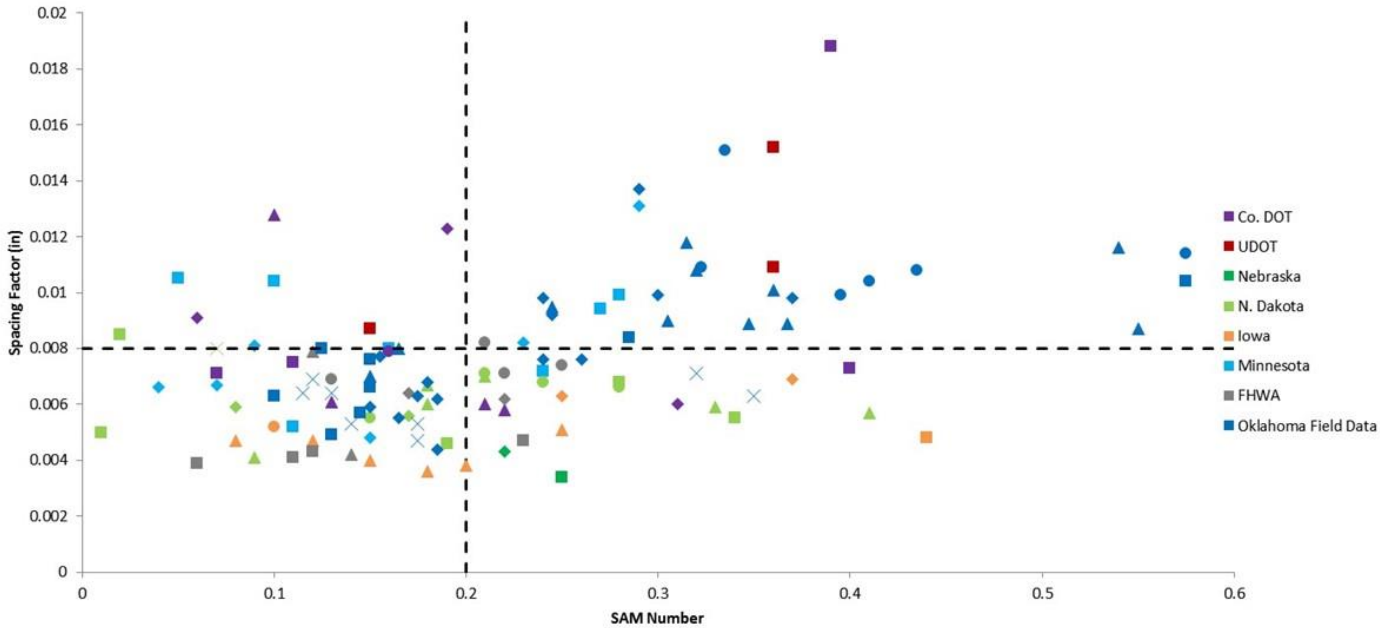


Figure 2 – Field testing data comparing the SAM number to the spacing factor for mixtures completed at OSU.

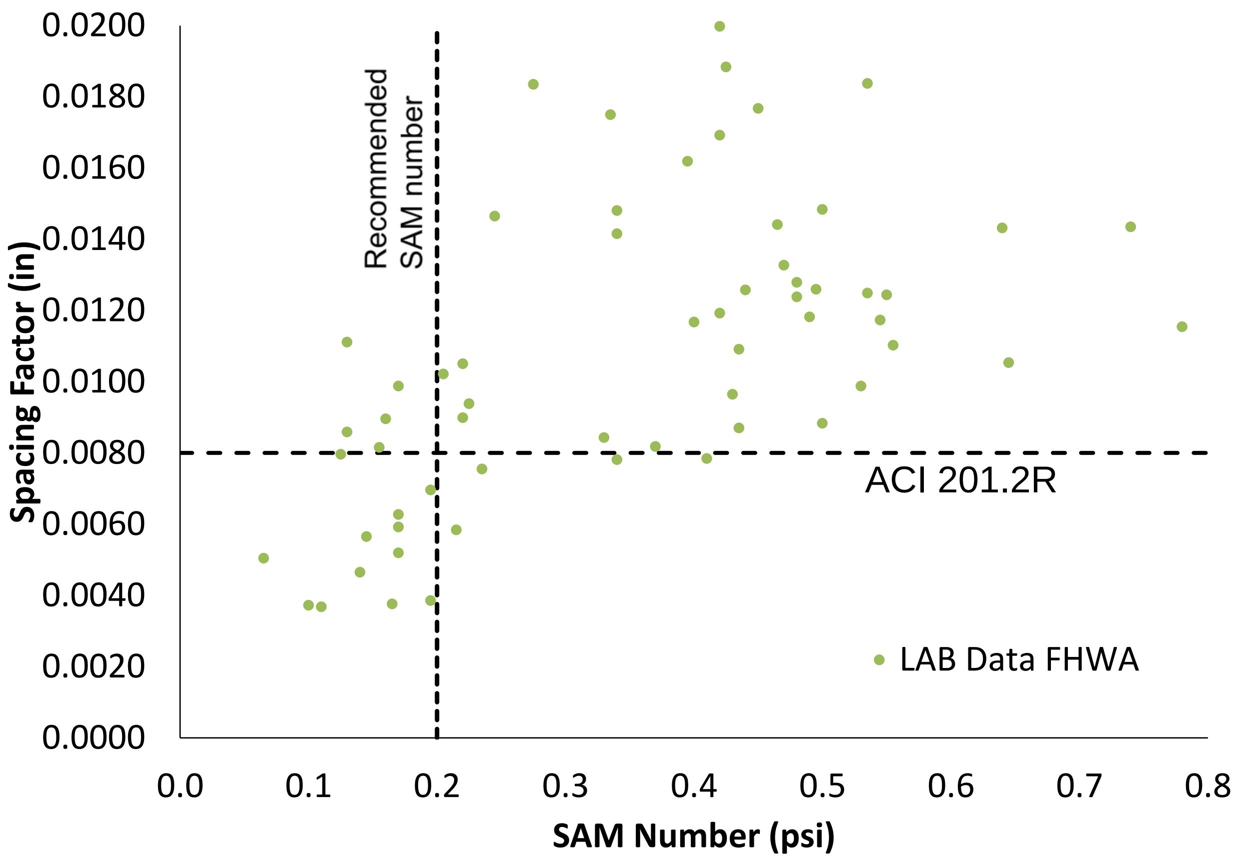


Figure 3 – Results from FHWA Turner Fairbanks Laboratory.

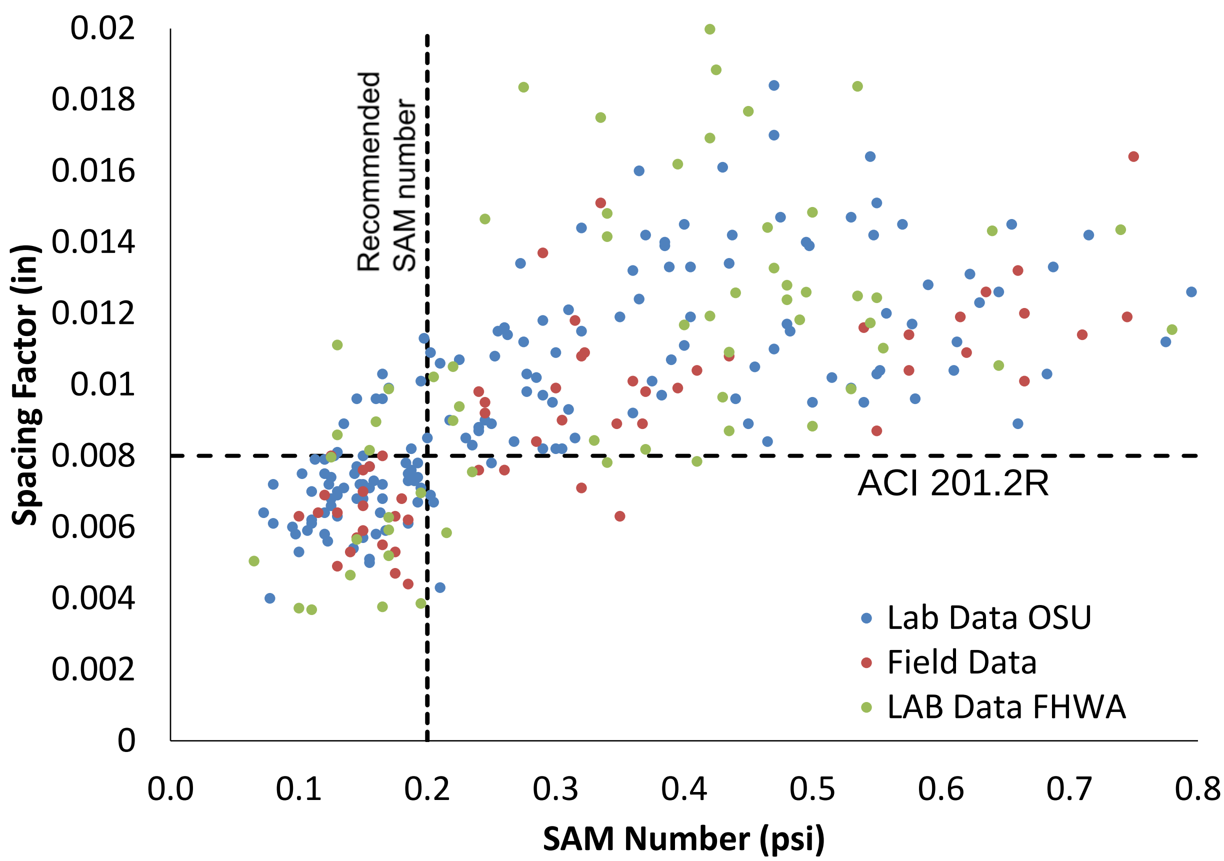


Figure 4 – A combination of the Figures 1, 2, and 3. This shows a comparison of the lab field, and 3rd party lab data.

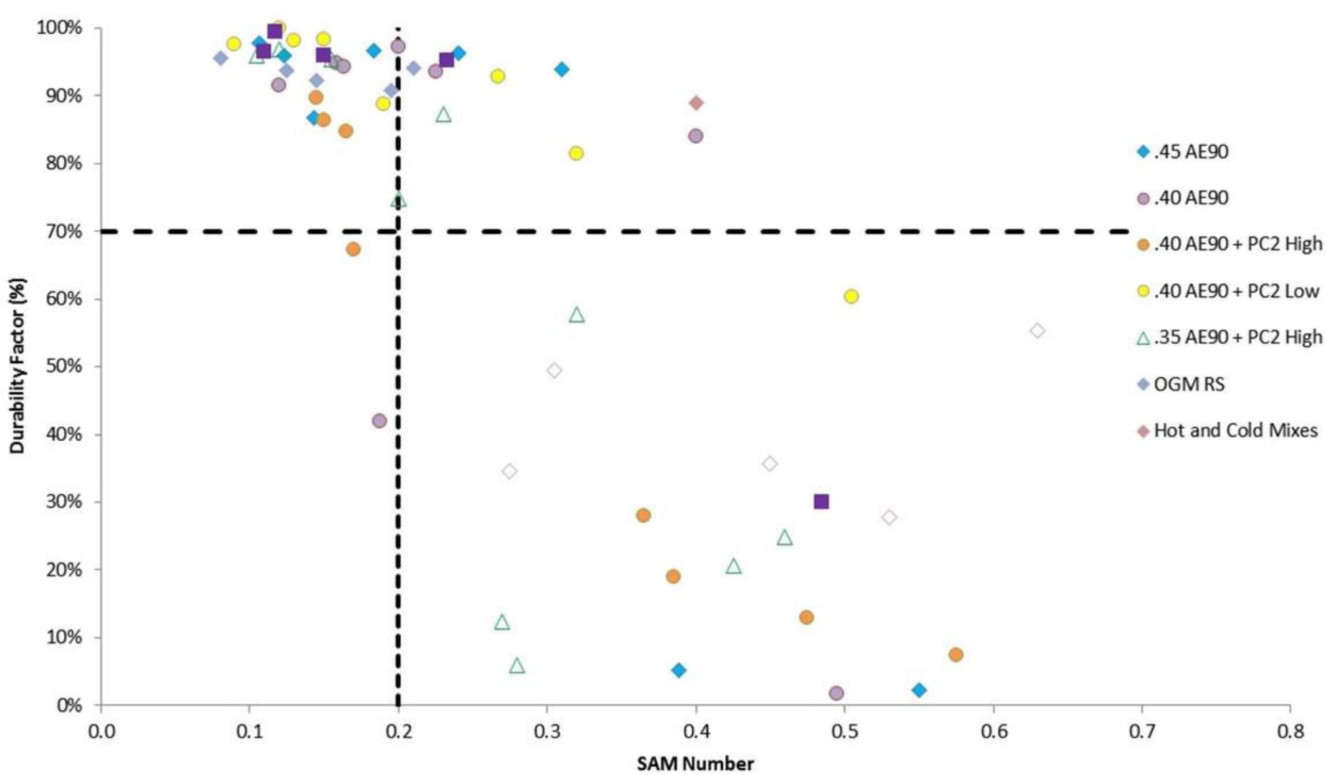


Figure 5 – The SAM number versus the Durability Factor from the C666 testing.

SORPTION (ASTM 1585)

1. CUT SAMPLES

- CUT OFF TOP 3/4" OF EACH CYLINDER

- CUT 2 SAMPLES- 2" THICK, 4" DIAMETER FROM EACH

- SAVE TOP AND BOTTOM SCRAP PIECES

- LABEL EACH SAMPLE WITH "SORP-MIXID"

2. MASS EACH SAMPLE

3. CONDITIONING

- PLACE 2 SORPTION SAMPLES AT 50%, PLACE 2 SAMPLES AT 80% RH

-MONITOR MASS EVERY 15 DAYS. REMOVE WHEN MASS CHANGES BY LESS THAN 0.02% (OR AS LONG AS TIME PERMITS)

4. TEST RESISTIVITY

4. ASTM 1585 (BEGINNING WITH PROCEDURE)

5. TEST RESISTIVITY, MASS

6. OVEN DRY, MASS

7. VACUUM SATURATE, MASS

No separate sample needed

ABSORPTION/ DESORPTION (DVS)

1. CUT SAMPLES

- DVS SAMPLES (0.8MM SLICES) WILL COME FROM SCRAP FROM DRYING TEST (MIDDLE MOST PORTION)

2. PLACE A LABEL ON IT WITH "DVS-MIXID"

3. DVS PROCEDURE

DEGREE OF SATURATION + POROSITY

1. DEMOLD

2. MASS ("SEALED MASS")

3. OVEN DRY, MASS

4. CUT

- 2 SAMPLES, 2" THICK, 4" DIAMETER

- LABEL EACH SAMPLE WITH "DOS-MIXID"

-USE REMAINING MIDDLE PIECE FOR DVS

5. MASS EACH SAMPLE

6. OVEN DRY, MASS EACH SAMPLE

7. VACUUM SATURATE EACH SAMPLE

8. MASS EACH

9. CALCULATE D.O.S.

10. CALCULATE POROSITY (ASTM 642)

- USE VACUUM SATURATION INSTEAD OF BOILING

11. UNIAXIAL RESISTIVITY

RESISTIVITY

1. LABEL EACH CYLINDER WITH "ρ-MIXID"

2. MEASURE RESISTIVITY ON SEALED CONDITION

3. ONE CYLINDER WILL BE USED TO TEST DOS AT THE CURRENT MATERIAL AGE

4. ONE CYLINDER WILL BE PLACED IN A CONDUCTIVE SOLUTION FOR 1 WEEK, OVEN DRY, MASS

5. ONE CYLINDER WILL BE PLACED IN A CONDUCTIVE SOLUTION FOR 2 MONTHS, OVEN DRY, MASS

6. RESISTIVITY AND MASS MEASUREMENTS WILL BE TAKEN PERIODICALLY FOR EACH SAMPLE.

7. MOISTURE CONTENT, DOS, MASS, AND RESISTIVITY WILL BE RECORDED WITH TIME

AE-LGCC

1. CORE EACH CYLINDER TO 2.25" DIAM (2.5" BIT)

2. CUT

- REMOVE TOP 3/4 "

-CUT 3 SAMPLES WITH 2" THICKNESS FROM EACH CYLINDER

-SAVE REMAINING ~3/4" BOTTOM PIECE AS SCRAP

3. LABELWITH "LGCC-MIXID"

4. OVEN DRY, VACUUM SATURATE TO 100% WITH LIME WATER

5. DRY EACH SAMPLE TO DESIRED LEVEL OF SATURATION

-75%, 80%, 85%, 90%, 95%, 100% (lower levels if we decide to use the leftover cylinder for this purpose)

- SEAL FOR 1-2 WEEKS

6. TEST RESISTIVITY

7. RUN IN LGCC-AE

8. TEST RESISTIVITY

DRYING

1. CUT

- FROM CYLINDER 1, REMOVE TOP INCH, CUT 1 50MM SAMPLE, CUT 1 10MM SAMPLE, CUT ANOTHER 50MM SAMPLE, SAVE BOTTOM AS SCRAP FOR DVS SAMPLE

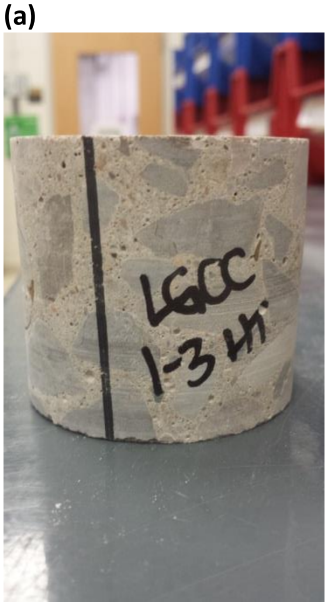
- FROM CYLINDER 2, REMOVE TOP 2 INCHES, CUT 1 10MM SAMPLE, CUT 1 50MM SAMPLE, CUT ANOTHER 10MM SAMPLE, SAVE BOTTOM AS SCRAP

-LABEL EACH WITH "DRY-MIXID"

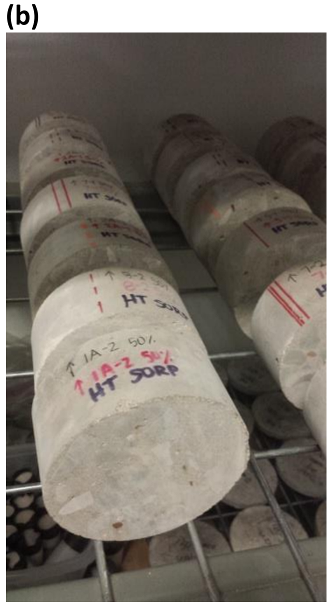
2. FOLLOW STADIUM TEST PROCEDURE (NOT DIFFUSION PORTION), BUT WITH VACUUM SATURATION NOT BOILING

Figure 6 - Sample cutting, conditioning, and testing plan for each series of mixtures

(a)



(b)



(c)

Figure 7 - Current Testing States (a) Sorption samples conditioning at 50% relative humidity (b) Sample labeling conventi a cut and cored LGCC sample (c) LGCC Test Setup

Mix	Fresh Air %	SAM Number	Mix ID	DOS	Projected LGCC Testing
0.45 AE90 Mix 5	6.58	0.107	5-3 #1	99%	COMPLETE
			5-3 #2	95%	
			5-4 #2	88%	
0.45 AE90 Mix 7	2.59	0.55	7-3 #1	98%	COMPLETE
			7-3 #2	92%	
			7-4 #2	90%	
0.45 AE90 Mix 2	3.88	0.24	2-3 #1	100%	COMPLETE
			2-3 #2	95%	
			2-3 #3	90%	
0.40 AE90 Mix 5	1.97	0.495	5A-3 #1	100%	COMPLETE
			5A-3 #2	95%	
			5A-3 #3	90%	
0.40 AE90 Mix 7	3.65	0.2	7A-3 #1	100%	COMPLETE
			7A-3 #2	95%	
			7A-3 #3	90%	
0.40 AE90 +575 Mix 2	7.2	0.17	2B-3 #1	100%	Apr-15
			2B-3 #2	95%	
			2B-3 #3	90%	
0.40 AE90 +575 Mix 1	4.31	0.385	1B-3 #1	100%	May-15
			1B-3 #2	95%	
			1B-3 #3	90%	
0.40 AE90 +575 Mix 8	2.44	0.575	8B-3 #1	100%	May-15
			8B-3 #2	95%	
			8B-3 #3	90%	
0.35 AE90 +575HIGH Mix 5	4.04	0.32	5C-3 #1	100%	Jun-15
			5C-3 #2	95%	
			5C-3 #3	90%	
0.35 AE90 +575HIGH Mix 7	9.24	0.155	7C-3 #1	100%	Jul-15
			7C-3 #2	95%	
			7C-3 #3	90%	
0.35 AE90 +575HIGH Mix 11	2.42	0.46	11C-3 #1	100%	Aug-15
			11C-3 #2	95%	
			11C-3 #3	90%	

Table 1 - Projected LGCC Freeze- Thaw Testing Schedule

Anticipated work next quarter:

The teams will continue preparing concrete mixtures to be investigated with the SAM and processing the materials produced previously. The ASTM C 666 testing will continue as well as the ASTM C 457 sample preparation for the samples provided by other states.

The team also plans to begin examining the rate of looking the absorption and desorption, rate of damage, and degree of saturation level on the damage with the concrete provided by OSU.

Significant Results:

The data from over 300 different laboratory and field mixtures completed by two different labs suggest that a SAM number of 0.20 can correctly determine if the spacing factor is above or below 0.008” about 93% of the time. There is also over 80% agreement between the SAM results and the ASTM C666 results.

A presentation on the progress of the project was given at the NCC meeting in Omaha, NE. Many of the sponsoring states were at the meeting and were able to be updated on the progress. In addition, webinars were given by Dr. Ley to the ACPA and their members, Missouri Science and Technology, North Dakota ACPA, Kansas KAPA, Utah ACPA, Iowa P conference, Colorado ACPA, Wisconsin ACPA, and the New Mexico Concrete School. Live presentations were made at th Wisconsin ACPA meeting, National ACI Convention, and NCC in Reno. Talks will also be given at the Minnesota Concrete Association. Meetings were held with the FHWA Mobile Concrete Lab, and Michigan DOT to discuss about the SAM progr

The implementation of an AASHTO Provisional Test Method is great progress.

Circumstance affecting project or budget. (Please describe any challenges encountered or anticipated that might affect the completion of the project within the time, scope and fiscal constraints set forth in the agreement, along with recommended solutions to those problems).

There was some delay getting the contracts signed. This has delayed the start of the project some but the research team doing their best to make up for this. The project is about three months behind schedule. A contract extension will likely be necessary.

Other than this issue the project is on time and on scope.

Potential Implementation:

A preliminary test method for the Super Air Meter has been presented to the AASHTO SOM. There was discussions at the AASHTO SOM meeting in Minneapolis and it appears that the test method will be approved in the fall and become availabl This is a great accomplishment. Work will continue on the project to develop the precision and bias statement.

One paper has been submitted to a conference and several more are being authored to submit soon.